IMMOBILIZED FILTERS FOR AIR FILTRATION

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This study will determine the feasibility of creating an immobilized bed of adsorbent particles using adhesives. The immobilized adsorbent will be designed for gas phase filtration in military respirators, in order to provide enhanced moldability (settling performance) and attrition resistance. The fabricated filter samples will be analyzed in order to determine the physical and chemical factors affecting mechanical strength and chemical filtration.

Five different resin systems were studied in this work; epoxy/amine, vinyl-ester, water-borne epoxy/amine and two water-borne urethane pre-polymer. The first two resins are organic but epoxy/amine system is more hydrophilic than vinyl-ester. Samples were cured through temperature cycles appropriate for their corresponding resin. Optical microscopy was used to look at the samples and determine the extent of dispersion of resin with the activated carbon particles. Compressive and flexural properties of samples of activated carbon particles with water-borne resin systems were evaluated. This effort was focused on distinguishing between brittle failure and ductile failure of the samples.

INTRODUCTION

Gas phase, adsorption based separation processes are usually conducted in cylindrical vessels under axial or radial flow. This symmetry offers several advantages in filter design. Restraining of the granular adsorbent material is facilitated because only one spring plate is required. Also uniform flow patterns are established. Cylindrical filter bed designs are employed almost exclusively in high pressure adsorber vessels because stresses are evenly distributed. Personal protective mask canisters and ventilation filters on the other hand are typically operated at ambient pressure allowing non-cylindrical designs to be considered. Respirator canister designs have been developed which have a reduced profile. Such filters would not be used as an external canister but rather as an integrated element, closely molded to the cheek or placed in some less cumbersome location.

If under rough handling or settling the adsorbent bed develops leak paths or regions of low density, premature breakthrough could result. An unrestrained bed can also result in increased particle attrition. When a unidirectional spring plate is no longer feasible, one solution would be to make the adsorbent structure self-supporting. Particles would be bonded together creating larger effective particles. High adhesive content in the interstices, on the surface or in the pores would result in additional mass transfer, and flow resistance and performance degradation relative to the un-immobilized material.

A recent patent, which specifically addressed the fabrication of filter canisters, concluded that dry mixing of adsorbent and binder worked best for small particles and wet mixing for large particles. Verification by microscopy was suggested. Polymer should be selected based on wetting ability, where more wetting of adsorbent would result in coating rather than contacting

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14. ABSTRACT

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the adsorbent. This work identified the best candidates as polyurethane, ethylene-vinyl acetate, and polyethylene. Binder particles sizes between 40 to 400 mesh were appropriate to minimize coating and pressure drop effects. One interesting observation was that samples prepared under compression could have high bulk densities as much as 110 percent of a loose packed bed of the same adsorbent.

The principal adsorbent considered here is a military specific material for chemical warfare protection, ASZM-T, which is an activated carbon, impregnated with metal oxides to promote detoxification of adsorbed chemical agents. This adsorbent is used for CW protection in a variety of filter sizes. The activated carbon adsorbent is formulated from microparticles, which are agglomerated with a petroleum, or pitch based binder. The short-range structural strength of the binder can be broken which would form dust. The cost advantage of granular ASZMT at $9/lb versus up to $200/lb for novel materials such as activated impregnated carbon cloth suggests that further use of the granular material is highly favored for disposable items such as respirator cartridges.

Performance properties of the adsorbent can be determined by a series of tests. Vapor filtration performance is measured by challenging the adsorbent under flow with both a strongly adsorbed simulant vapor, DMMP, to measure physical adsorption properties and a weakly adsorbed reactive vapor, cyanogen chloride (CK), to asses the activity of the chemical reaction based impregnant formulation. In addition, pressure drop across the filter and accumulated dust measurements are important performance parameters.

The ASZM-T utilizes an impregnant formulation of metal oxides to react with weakly adsorbed acid gases. The separation is accomplished by reaction between the toxic gas and metallic compounds (reference report) deposited into the pores and onto the surface of the particles. To enhance the physical adsorption of catalysts, triethylenediamine known as TEDA, was applied to the activated carbon particles. This limits the upper temperature for processing samples to approximately 80°C. Since the porosity of these particles greatly enhances their separation capacity, processing factors that affect porosity and/or accessibility of the pores are critical to the overall performance of the material.

The ASZMT material must be processed in a moderate temperature range. This limits the number of polymers that are possible to consider. The binder must contact the surface of the adsorbent but not coat it. The breakthrough testing provides a severe challenge to assess the extent of surface coverage.

Polymers are divided into two main categories; thermoplastics and thermosets. In thermoplastics, physical interactions and entanglements create the cohesion between polymer chains. In thermosets, chemical bonds between chains / crosslinks, in addition to physical bonding, create a network that cannot be significantly reshaped unless some chemical bonds are broken. Both thermoplastics and thermosets exhibit a softening temperature named glass transition temperature, Tg. Below Tg, both types of polymer are rigid / glassy. Above Tg, thermoplastics become liquid like; flow under their own weight, however, thermosets exhibit rubbery behavior. Thermoplastics can lose their shape, cohesion, and all their mechanical attributes when heated above their softening point while thermosets retain some of their useful properties

Most thermoset resin systems consist of a viscous liquid prepolymer and a hardner, whose mixture gels and solidifies upon heating. The liquid state of the mixture before curing allows for ease of processing of these materials. Therefore, the initial experiments were focused

on using thermosets as the adhesive for immobilizing the activated carbon particles. Moreover, some useful mechanical properties; such as elasticity, ductility, and fracture toughness of thermosets have wider range than those of thermoplastics do. Further mechanical advantages of thermosets will be discussed later in this report.

It is expected that low resin loading would have less adverse effect on the porosity and packing of the particles. Therefore, good dispersion of resin over activated carbon particles is desired. This implies that the resins should have low viscosity for effective mixing. For this reason, three water-borne resin systems were tested in this work. It is also desired that the organic resin not fill the micropores of activated carbon during processing. One approach is to fill the micropores with water; thus limiting the accessibility of organic molecules to the pores by taking advantage of the incompatible nature of organic and aqueous phases, and then to mix the particles with resin. To this end, activated carbon particles, both dry and saturated at 50°C and 85% relative humidity, were tested with five resin systems. Initial work will entail selecting the resin systems for evaluation, including both thermosets and thermoplastics. The potential exists for these organic materials to alter the adsorbent characteristics of the activated carbon. Water-borne systems are often used for coatings and in this case would limit the need for organic solvents and could potentially provide a method for limiting pore filling while still providing adequate bonding of particles. Thus emphasis will be placed on water-borne systems for this investigation.

This study will determine the feasibility of creating an immobilized bed of adsorbent particles using adhesives. The immobilized adsorbent will be designed for gas phase filtration in military respirators, in order to provide enhanced moldability (settling performance) and attrition resistance. The fabricated filter samples will be analyzed in order to determine the physical and chemical factors affecting mechanical strength and chemical filtration.

MATERIALS

Five different resin systems were studied in this work; epoxy/amine, vinyl-ester, water-borne epoxy/amine and two water-borne urethane prepolymer. The first two resins are organic but epoxy/amine system is more hydrophilic than vinyl-ester. Dry activated carbon particles and particles saturated at 50°C and 85% relative humidity were mechanically mixed with these resin systems at different loadings to determine the minimum required resin content for good adhesion. Samples were cured through temperature cycles appropriate for their corresponding resin. Optical microscopy was used to look at the samples and determine the extent of dispersity of resin with the activated carbon particles. Compressive and flexural properties of samples of activated carbon particles with water-borne resin systems were evaluated. This effort was focused on distinguishing between brittle failure and ductile failure of the samples. Samples cut out of cartridges produced by the 3M company were tested for their flexural strength and the results were compared to those obtained using the water-borne resins. The applicability of a spraying technique was tested using one of the water-borne urethane prepolymers sprayed on the activated carbon particles. Experiments were conducted to determine the effect of the viscosity of the resin on this technique.

RESULTS

Micrographs of the bonded samples can be instructive of the extent and efficiency of the contact. Figure 1 presents a 20X view of the un-immobilized adsorbent sample. Figure 2 presents

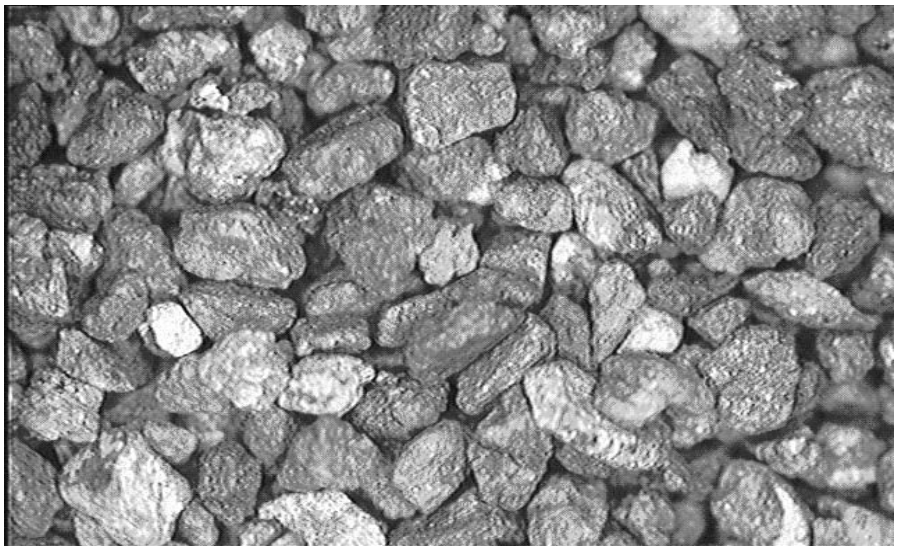

Figure 1. Activated carbon particles, ASZM-T, as received.

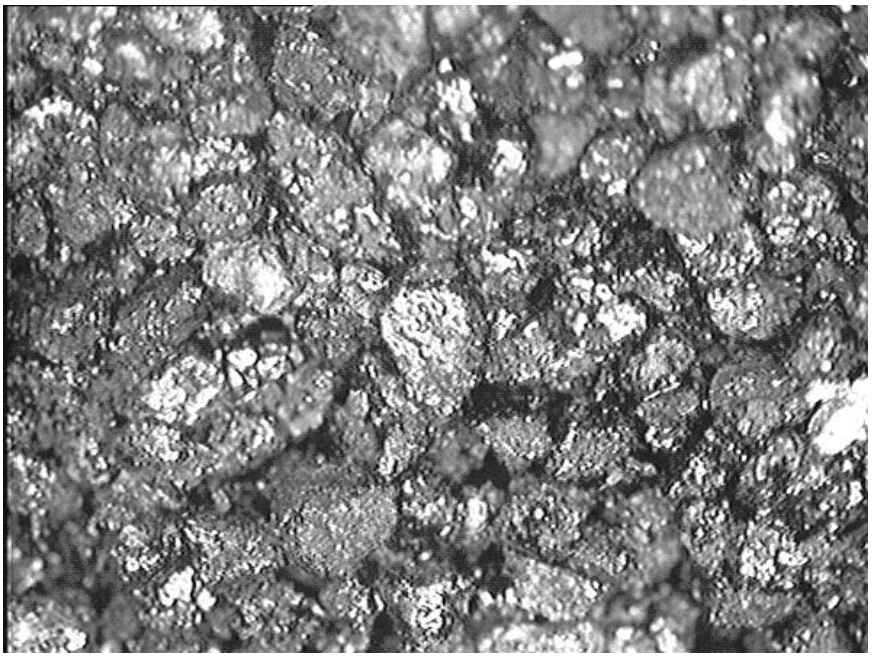

Figure 2. Sample of dry activated carbon with 10% water-borne epoxy/amine resin.

the results for a sample prepared with 5% water-borne epoxy/amine. The contact points between the particles are seen to be randomly oriented. Thus approaches which coat the surface are highly inefficient. Figure 2 indicates that the waterborne samples result is excessive coverage.

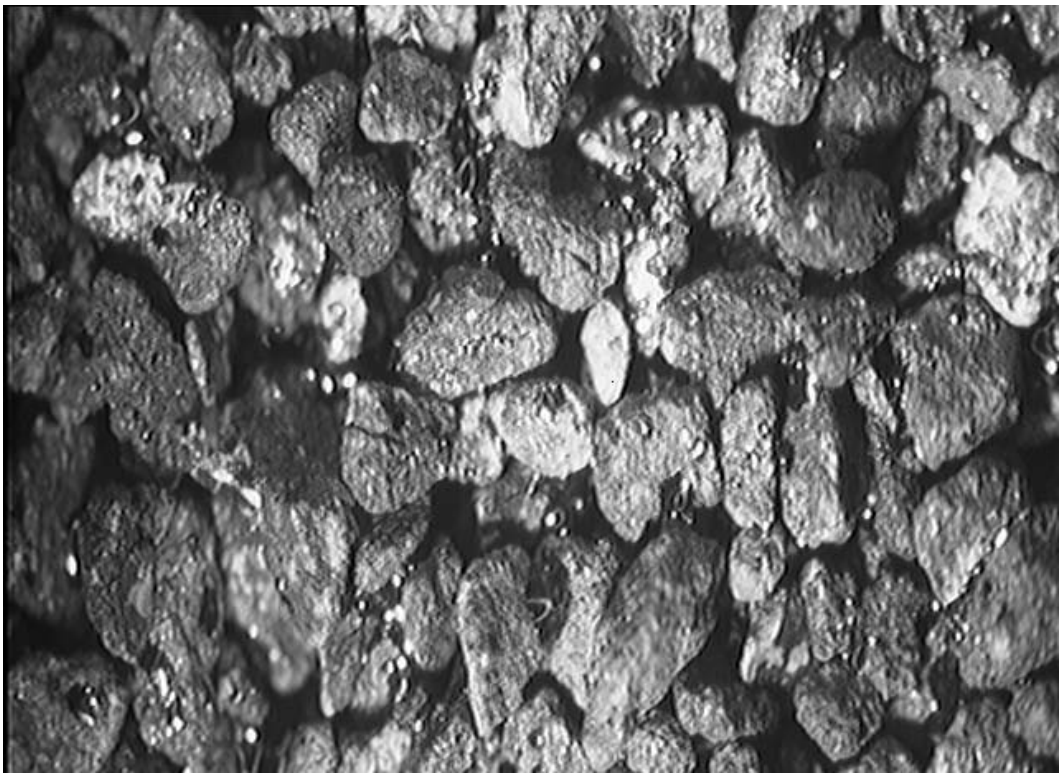

Figure 3. Sample of dry activated carbon with 15% cured 50 mesh thermoplastic.

It can be seen that a sample bonded with granular adsorbent produces less surface coverage than waterborne samples. The effect of bed packing is seen in figure 4. A 30% decrease in breakthrough time results from a loosely packed bed due to tamping.

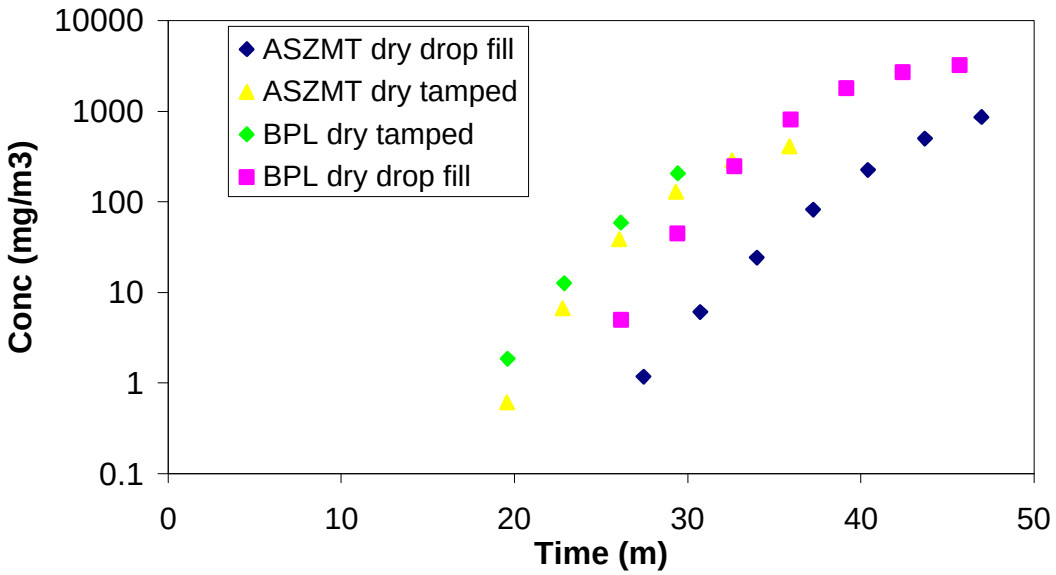

Figure 4. Sample of dry activated carbon with 15% cured 50 mesh polyester.

The mechanical properties of immobilized beds were assessed using three techniques: stress/strain, compression and attrition. Figure 5 illustrates the effect of loading on compressive strength. Epoxy samples are stronger in compression while urethane samples give better flexural properties. Attrition effects are measured by weight loss on a vibration table.

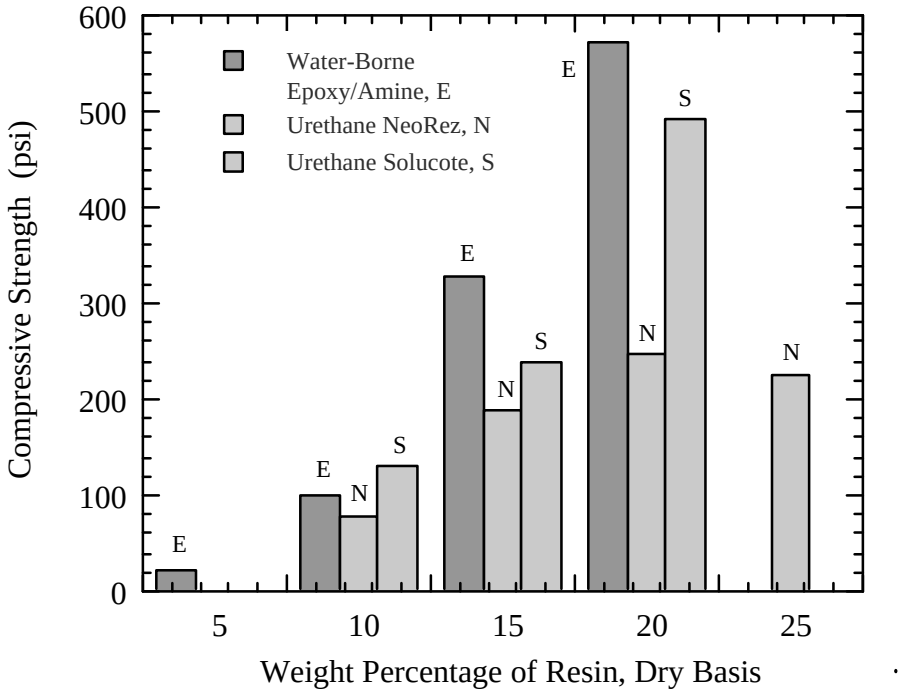

Figure 5. Comparison of compressive strength versus resin content for samples of dry activated carbon with water-borne epoxy/amine, urethane Solucote and urethane NeoRez.

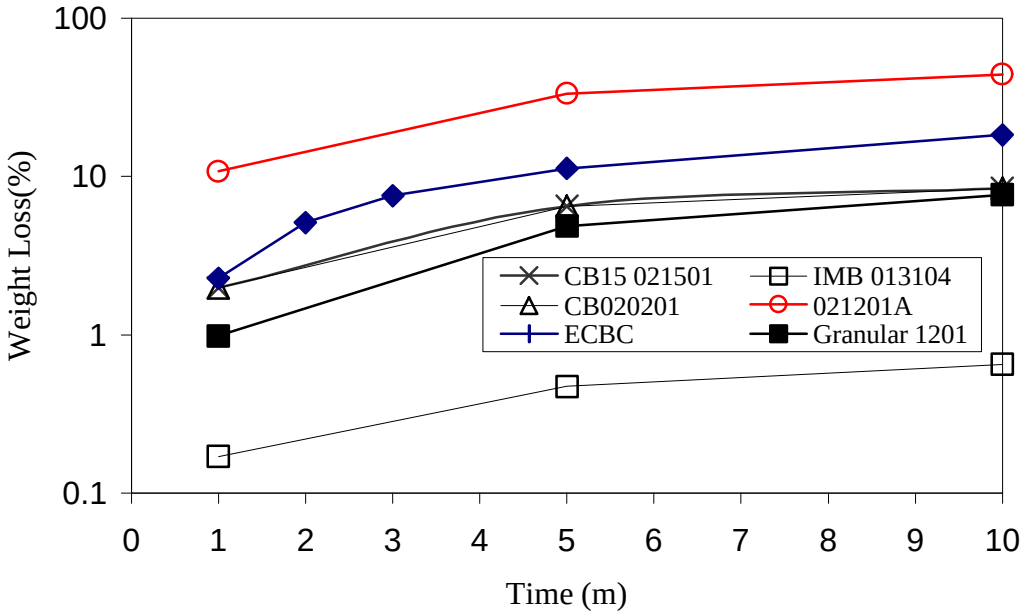

Figure 6. Attrition weight loss for several immobilized samples at 15% loading.

The change in rate of attrition is demonstrated in figure 6. The initial rate is affected by loose granules and edge effects. This rate decreases with time.

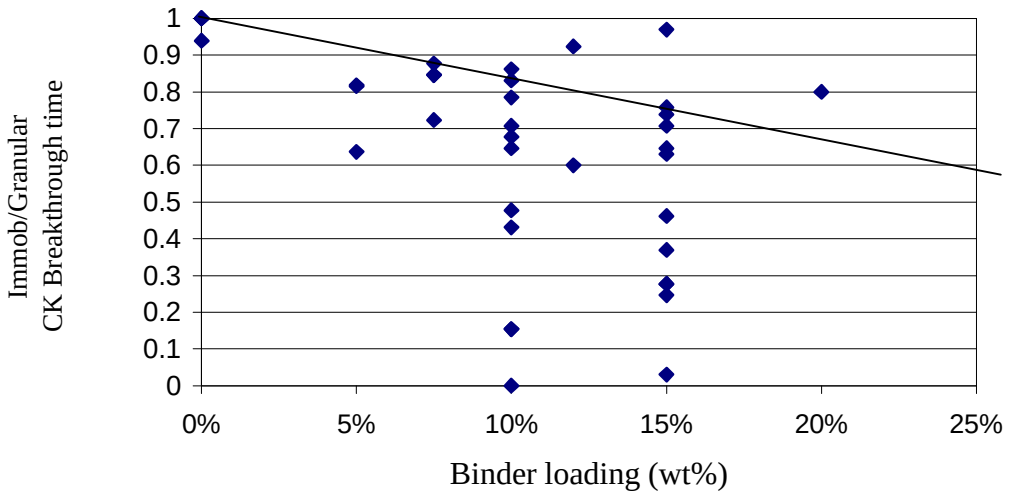

Figure 7. Effect of loading on breakthrough behavior.

The breakthrough results for bonded samples are presented in figure 7. Despite the large scatter in the data there appears to be a linear trend the breakthrough time reduction with increasing loading. The increase in bonding strength as related to this filtration reduction must be addressed by some optimization scheme.

CONCLUSIONS

Immobilized samples of granular activated carbon have been fabricated with strong mechanical properties, attrition resistance and light gas filtration performance loss of 10-20%. The wet techniques using waterborne epoxy and urethane result in significant surface coverage with associated loss of filtration performance.

CITATIONS

1. Mahle, J.J., Palmese, G., Ziaee, S., “Filter Immobilization Studies Part 1. Polymer Screening”, U.S. Army, APG MD, ECBC-TR-060, (1999).